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NAREL STANDARD OPERATING PROCEDURE

ANION ANALYSIS

FOR THE

$PM_{2.5}$ CHEMICAL SPECIATION QA PROGRAM

PM2.5/SOP-2

Monitoring and Analytical Services Branch
National Air and Radiation Environmental Laboratory
Office of Radiation and Indoor Air

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PM2.5/SOP-2

REVISION HISTORY

Rev.	Responsible Official	Date
0	Steve Taylor	2000-11-06

1.0 PURPOSE

1.1 This document describes the procedures used at the National Air and Radiation Environmental Laboratory (NAREL) for the extraction and subsequent determination of selected anions in $PM_{2.5}$ filter extracts by ion chromatography.

2.0 SCOPE AND APPLICATION

2.1 This document provides detailed instructions for sample preparation and analysis of air filters by ion chromatography. Procedures for sample inspection, measurement, calculation, documentation, archival, safety, and quality control are included.

2.2 This method is restricted for use by or under the supervision of analysts trained in the operation of the Dionex model DX 500 ion chromatograph. Each analyst must demonstrate the ability to generate acceptable results with this method.

3.0 DEFINITIONS

3.1 **CCB** - Continuing calibration blank.(reagent blank)

3.2 **ICV** - Instrument calibration verification standard. An independent standard that is derived from a second source.

3.3 **CCV** - Continuing Calibration Verification. A quality control check standard which is analyzed periodically to test the accuracy of the measurement system.

3.4 **COC** - Chain Of Custody. An unbroken trail of accountability that ensures the physical security of samples, data, and records.

3.5 **I.S.** - Internal Standard. A component added to the sample before analysis to provide relative retention times and relative response factors for data reduction.

3.6 **LCS** - Laboratory Control Sample. A quality control sample that contains spiked analytes which is prepared and analyzed along with each sample batch.

3.7 **MASB** - Monitoring and Analytical Services Branch. The branch at NAREL is responsible for the analysis of environmental samples for radioactive and/or mixed waste contamination.

3.8 **NAREL** - National Air and Radiation Environmental Laboratory.

3.9 **NIST** - National Institute of Standards and Technology.

3.10 **$PM_{2.5}$** - Particulate Matter with an aerodynamic diameter less than or equal to 2.5 micrometers.

3.11 **PTFE (Teflon®)** - Polytetrafluoroethylene manufactured and marketed by Dupont

using its “Teflon” registered trademark.

3.12 **QAC** - Quality Assurance Coordinator. The person with primary responsibility for overseeing the NAREL QA/QC Program.

3.13 **RTI** - Research Triangle Institute, the independent contractor laboratory for the $PM_{2.5}$ Chemical Speciation Program.

3.14 **SHEM** - Safety Health and Environmental Management

3.15 **SOP** - Standard Operating Procedure. The officially approved document that describes in detail the steps of a procedure for performing a routine or repetitive task.

4.0 SUMMARY OF METHOD

4.1 Filters are extracted in reagent water by sonication and mechanical shaking. A small volume of extract typically 100 µL is eluted through an ion exchange column where the anions are separated by their different affinities for the active resin sites in the separator column. A suppressor column is used to reduce the eluent background, while at the same time converting the target anions to a form that is more conductive. Species are detected by their conductivity and are identified and quantified by comparison to calibration standards.

5.0 INTERFERENCES

5.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of anions eluting close to the anions of interest will result in an interference. Sample dilution, changing eluent strength, and/or flow rate can be used to solve most interference problems associated with retention times.

5.2 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

5.3 All liquids should be filtered before they are introduced to the chromatographic system to prevent damage to instrument pumps, check valves, and columns.

6.0 EQUIPMENT AND SUPPLIES

6.1 Ion chromatograph (Dionex Model DX-500) consisting of:

6.1.1 LC20 chromatography module

6.1.2 IP25 Isocratic pump

6.1.3 CD20 conductivity detector

6.1.4 AS40 autosampler

6.1.5 Conductivity cell

6.1.6 PeakNet® 5.1 control software

6.1.7 IonPac AG12A 4-mm guard column

6.1.8 IonPac AS12A 4-mm analytical column

6.1.9 Suppressor, ASRS-ULTRA 4-mm

6.1.10 Eluent reservoirs pressurized with helium

6.1.11 Dell PII 450 MHz with Windows 95

6.2 Centrifuge tubes 50-mL

6.3 Pipettors

6.4 Ultrasonic bath with plastic test tube rack for 50-mL tubes

6.5 Mechanical shaker

6.6 Volumetric flasks

6.7 Dionex autosampler vials with filter caps

6.8 Nylon filters (Gelman Nylasorb® , 47-mm diameter, 1-µm pore size, Product no. 66509)

7.0 Reagents

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

7.2 Reagent water with a specific resistance of 18.0 Mohm-cm or better should be passed through a 0.2-µm filter before use.

7.3 Fluoride, 1000 ppm solution , VWR catalog # VW3406-2 or equivalent.

7.4 Chloride, 1000 ppm solution, VWR catalog # VW3396-2 or equivalent.

7.5 Sodium Nitrite, VWR/J.T. Baker catalog # JTP684-7 or equivalent. A 1000 ppm Nitrite solution is prepared by adding 0.150 g of the neat material to a 100-mL volumetric flask and fill to the mark with reagent water.

7.6 Nitrate, 1000 ppm solution, SPEX catalog # AS-NO39-2Y or equivalent.

7.7 Sulfate, 1000 ppm solution, SPEX catalog # AS-SO49-2Y or equivalent.

7.8 Sodium selenite, Aldrich catalog # 48181-5 or equivalent.

7.9 Sodium selenate decahydrate, Aldrich catalog # 450294-5G or equivalent.

7.10 Sodium Carbonate anhydrous powder(Na_2CO_3), Fisher catalog # S263 or equivalent.

7.11 Sodium Bicarbonate powder($NaHCO_3$), Fisher catalog # S233 or equivalent.

7.12 Eluent Stock solution, 45mM $NaHCO_3$/400mM Na_2CO_3. Dissolve 0.378 g of $NaHCO_3$ and 4.240 g of Na_2CO_3 in 100 ml of reagent water.

7.13 Eluent solution, 0.45mM $NaHCO_3$/4.00mM Na_2CO_3. Add 10 mL of eluent stock solution to a 1000-mL volumetric flask and fill to the mark with reagent water.

7.14 Internal Standard solution, 1000 ppm. Dissolve 0.136 g of Sodium Selenite (Na_2SeO_3), 0.258 g of sodium selenate decahydrate ($Na_2SeO_4 \cdot 10\ H_2O$),0.378 g of $NaHCO_3$ and 4.240 g of Na_2CO_3 in reagent water and dilute to 100 mL. This internal standard solution contains selenite at 1000 µg/mL, selenate at 1000 µg/mL, and is in 45 mM $NaHCO_3$/400 mM Na_2CO_3.

7.15 Mixed Anions standard. Using 1000 ppm stock solutions and a 100-mL volumetric flask, prepare a mixed anions standard in reagent water as shown in table 1.

Table 1

Original Stock	Initial concentration	Volume added to 100-mL flask	Final concentration
Fluoride	1000 ppm	1 mL	10 ppm
Chloride	1000 ppm	1 mL	10 ppm
Nitrite	1000 ppm	1 mL	10 ppm
Nitrate	1000 ppm	2 mL	20 ppm
Sulfate	1000 ppm	2 mL	20 ppm

7.16 Using the mixed anions standard, prepare the working standards with reagent water in 100-mL flasks as shown in table 2.

Table 2

Standard	Volume (mL) of Mixed Stock per 100 mL of Working Standard Prepared	Final Concentration of F, Cl, and NO_2 (ppm)	Final Concentration of NO_3 and SO_4 (ppm)
1	0.2	0.02	0.04
2	1	0.10	0.20
3	2	0.20	0.40
4	5	0.50	1.00
5	10	1.00	2.00
6	15	1.50	3.00
7	20	2.00	4.00

7.17 A multi-analyte standard must be prepared from different lot numbers than those used for calibration. This Initial Calibration Verification (ICV) standard must be analyzed at each initial calibration to gain confidence in the accuracy of each calibration curve.

7.18 A consistent technique must be used for adding Internal standard (I.S.) solution to standards and to sample extracts. See the procedure section of this SOP for instructions concerning the addition of internal standard solution to all samples immediately before analysis.

8.0 SAFETY

8.1 All procedures performed at NAREL must be conducted following the requirements detailed in the *NAREL Chemical Hygiene Plan* and the *NAREL Radiation Safety Manual.*

8.2 All NAREL laboratory personnel are expected to use good laboratory practices. Most of the safety training is provides by the SHEM officer. All lab personnel are expected to conform to directives given by the SHEM officer.

9.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

9.1 Because of its QA role within the $PM_{2.5}$ Chemical Speciation Network, NAREL will analyze samples that are split with the independent contract laboratory, Research Triangle Institute (RTI). NAREL will work closely with RTI to coordinate its QA activities

with the on-going laboratory services that RTI provides to the program. The sequential events in the life cycle of a filter analyzed at NAREL are described as follows.

9.1.1 Filters are purchased by NAREL

9.1.2 A representative number of filters from each lot is inspected for defects and tested for contamination.

9.1.3 Filters are cleaned if necessary.

9.1.4 Filters are given a unique identification number.

9.1.5 Filters are shipped to RTI with chain-of-custody (COC) documentation.

9.1.6 Each filter is installed in the appropriate filter module at RTI.

9.1.7 The filter and module assembly is shipped to the field site by RTI.

9.1.8 At the field site, a duplicate air sample is collected onto the filter using either a spare channel available on the sampler unit or using a co-located sampler unit.

9.1.9 The duplicate sample is shipped to NAREL from the field site.

9.1.10 At NAREL, the filter is removed from its module.

9.1.11 Filters are stored in a freezer until extraction and analysis.

9.1.12 The exposed filter is extracted and analyzed.

9.1.13 The empty filter module is shipped back to RTI with a COC.

9.1.14 Filters must be extracted within 20 working days of sample receipt at the laboratory.

10.0 PROCEDURE

10.1 New filter lots received at NAREL will be logged into a filter inventory database used to track the activities of each individual filter. Additional testing, beyond that already completed by the manufacturer, will be performed as follows.

10.1.1 At least three filters from each new lot will be extracted and analyzed at NAREL. If target analyte is detected at a level above the report limit of a method blank, then every filter in the lot must be cleaned, dried, and re-packaged before it can be used for field sampling.

10.1.2 Each individual filter will be examined using a light box and magnifying lens for visual signs of manufacturing flaws. A filter will be rejected for use in the program if it contains any of the following defects.

10.1.2.1 Pinholes.
10.1.2.2 Separation of ring
10.1.2.3 chaff or flashing
10.1.2.4 loose material
10.1.2.5 discoloration
10.1.2.6 filter nonuniformity
10.1.2.7 any other observed defect which might degrade filter performance

10.2 Filter Extraction Procedure

10.2.1 Remove filters from the freezer and allow them to equilibrate to room temperature.

10.2.2 Using gloved hands and tweezers, place filter in a 50-mL plastic centrifuge tube that has been labeled with the filter ID number. Be sure to place the label near the top of the tube so that it will not be lost during the sonication step.

10.2.3 Place a clean unused filter into a 50-mL tube and label this tube as the laboratory method blank.

10.2.4 Place a clean unused filter into a second 50-mL tube and label this tube as the Laboratory Control Sample. Spike this sample with 2.0 mL of the mixed anions standard.

10.2.5 Label a 50-mL tube as the laboratory reagent blank.

10.2.6 Since filters will be analyzed for both anions and cations, the extraction solvent will be reagent water. Except for the LCS, dispense 25.0 mL of reagent water into each tube. Dispense 23.0 mL of reagent water into the LCS tube.

10.2.7 Ensure that each filter is completely submerged in the reagent water. Use a Teflon® rod to secure if necessary.

10.2.8 Place the batch of centrifuge tubes into a plastic rack and place the rack into the ultrasonic bath so that the water level of the bath is higher than the fluid level in the extraction tubes. Sonicate for 60 minutes.

10.2.9 Monitor the bath temperature during sonication. Do not allow the temperature to exceed 27 °C. Ice can be used to reduce the bath temperature.

10.2.10 Secure the rack to the shaker table and shake overnight in a cold room (4°C) at approximately 60 cycles per minute. Make sure that the tubes are capped securely to prevent leakage. The rack should be should be placed on its side during shaking. A piece of cardboard fastened to the rack with rubber bands will prevent the tubes from falling out.

10.2.11 Complete the forms in the extraction log book.

10.2.12 Store the extracts in a refrigerator at 4°C until analysis.

10.3 IC Calibration Procedure

10.3.1 Initialize the instrument operating conditions from the PeakNet® software. Allow the pump pressure and background detector signal to stabilize. Record the pressure and background conductivity in the anions logbook.

10.3.2 Create a sample schedule with the PeakNet® software by entering the appropriate information for each injection such as sample name, method, filename, and other information necessary for method calibration and sample calculations. The naming convention for a raw data file follows.

Ammddyya_###.dxd

(example: c:\peaknet\data\anions\A070400a\A070400a_001.dxd for the first file acquired in the first schedule on July 4, 2000)

10.3.3 The naming convention establishes a base for each filename that begins with an “A” for Anions, followed by six characters expressing the date of the schedule, and ends with an “a” for the first schedule of the day, a “b” for the second schedule of the day, and so forth. The underscore character “_” separates the base of the filename from three numerical characters assigned by the software starting with 001 for the first file, 002 for the second file, and so forth. Notice that a directory must be created below the pathway “c:\peaknet\data\anions\” to contain the schedule of raw data files, and this new directory should be given the eight character (base) name of the data files it will contain. Make the new directory the default pathway for the schedule.

10.3.4 The analytical sequence of injections are as follows: instrument blank (reagent water with I.S.), followed by the calibration standards from lowest to highest as listed in table 2, and ending with an Initial Calibration Verification (ICV) standard.

10.3.5 Prepare vials for the AS40 autosampler by first writing a label on each

vial, and then pipet 50 µL of internal standard solution into each vial. Pipet 5 mL of each standard into the appropriately labeled vial and install filter caps. Invert every vial several times to insure good mixing. After mixing, each sample has been spiked with internal standard to a consistent 10-ppm level and each sample has been spiked with NaOH to the 15 mM level.

10.3.6 Load the vials into cassettes and place the cassettes in the AS40 autosampler. Press the run button on the AS40 to advance the first sample into position.

10.3.7 Start the analysis by selecting "Run" from the PeakNet® main dialog box. Select "Run" from the menu and "Start" to begin the calibration.

10.3.8 After all standards have been analyzed, complete the anions logbook forms as described in Appendix B.

10.3.9 Optimize the method calibration using the PeakNet® optimize software. The correlation coefficient for the calibration curve must be ≥ 0.995 for each analyte.

10.3.10 After optimization, reprocess the calibration standards and the ICV as unknown solutions. The software must properly identify each analyte in each standard. Observe the calculated concentration of analyte. The calculated value of each analyte must be within ± 10% of the expected value except for the low-level standard which must be within ± RL of its expected value where RL is the Reporting Limit for the laboratory method blank.

10.3.11 Internal standards are mixed into every sample to monitor the success of data acquisition. The retention time of the internal standards should be monitored for drift. Failure of the software to identify an internal standard in the chromatogram indicates excessive retention time drift and requires corrective action.

10.4 IC Filter Analysis Procedure

10.4.1 Initialize the instrument operating conditions from the PeakNet® software. Allow the pump pressure and detector signal to stabilize. Record the pressure and background conductivity in the anion logbook.

10.4.2 Create a sample schedule with the PeakNet® software by entering the appropriate information for each injection such as sample name, method, filename, and other information necessary for sample calculations. The naming convention for a raw data file follows.

Ammddyya_###.dxd

(example: c:\peaknet\data\anions\A070400b\A070400b_001.dxd for the first file acquired in the second schedule on July 4, 2000)

10.4.3 The naming convention establishes a base for each filename that begins

with an “A” for Anions, followed by six characters expressing the date of the schedule, and ends with an “a” for the first schedule of the day, a “b” for the second schedule of the day, and so forth. The underscore character “_” separates the base of the filename from three numerical characters assigned by the software starting with 001 for the first file, 002 for the second file, and so forth. Notice that a directory must be created below the pathway “c:\peaknet\data\anions\” to contain the schedule of raw data files, and this new directory should be given the eight character (base) name of the data files it will contain. Make the new directory the default pathway for the schedule.

10.4.4 Remove the extraction tubes from the refrigerator and allow them to warm to room temperature. These extracts will be used for both the anions and cations analyses.

10.4.5 The analytical sequence of injections must start, continue, and end with a Continuing Calibration Verification (CCV) standard as follows.

instrument blank (recommended)
CCV
up to ten sample extracts
CCV
up to ten sample extracts
CCV
⋮
CCV

10.4.6 Prepare vials for the AS40 autosampler by first writing a label on each vial, and then pipet 50 µL of internal standard solution into each vial. Pipet 5 mL of sample or standard into a separate vial and install filter caps. Invert every vial several times to insure good mixing. After mixing, each sample has been spiked with internal standard to a consistent 10-ppm level and each sample has been spiked with NaOH to the 15 mM level.

10.4.7 Load the vials into cassettes and place the cassettes in the AS40 autosampler. Press the run button on the AS40 to advance the first sample into position.

10.4.8 If cations are not being analyzed at this time, insure that caps are secure on the 50-mL extraction tubes and return them to the refrigerator.

10.4.9 After all samples have been analyzed, complete the anions logbook forms.

10.4.10 The instrument calibration must be verified by analyzing CCVs within the analytical sequence. The software must properly identify each analyte in each CCV. Observe the calculated concentration of analyte in each CCV. The calculated value of each analyte must be within ± 10% of the expected value. Failure of any CCV requires corrective action that includes re-analysis of all extracts since the last CCV that passed criteria.

10.4.11 Internal standards are mixed into every sample to monitor the success of data acquisition. The retention time of the internal standards should be monitored for drift. Failure of the software to identify an internal standard in the chromatogram indicates excessive retention time drift and requires corrective action such as updating the center of the analyte search window. Library retention times may not be updated except to those values demonstrated by a CCV.

10.5 Calculation

10.5.1 Sample component identification and concentration is computed automatically by the Dionex PeakNet® software.

10.5.2 Examine the data at the end of the run. If any analyte concentration exceeds the calibration curve, dilute, and rerun the sample for that analyte.

10.5.3 Print those reports needed for data review and archival.

11.0 QUALITY ASSURANCE

11.1 Each new lot of filters are tested at NAREL for contamination and if necessary are cleaned to satisfy blank criteria before any filter from the lot is submitted to the field for sample collection. Each individual filter is visually inspected for manufacturing or handling flaws using a light box and a magnifying lens before it is released for sample collection.

11.2 Exposed filters must be stored in a freezer until extracted. Temperature in the freezer is monitored and recorded in a logbook.

11.3 Filters must be extracted and analyzed within 20 work days of sample receipt at the laboratory.

11.4 Internal standards are used to monitor the success of every injection at the instrument.

11.5 No samples may be analyzed until an acceptable calibration curve has been established and verified by an independent standard (ICV) prepared from different lot numbers. If the analyte concentration determined for the ICV differs by more than 10% from its expected value, a new initial calibration must be established.

11.6 The analysis of every filter extract must be bracketed by a successful CCV. Failure of any CCV requires corrective action that includes re-analysis of all extracts since the last CCV that passed criteria.

11.7 Field blanks are analyzed to monitor for overall positive bias reported for field samples.

11.8 A laboratory method blank is analyzed with each batch of filters to monitor for positive bias created by laboratory contamination. If the analyte of interest is detected in

the method blank, any sample in which the analyte is present at less than 10 times the level detected in the blank must be re-analyzed.

11.9 Reagent blanks are routinely analyzed to monitor for sources of laboratory contamination.

11.10 A Laboratory Control Sample (LCS) must be extracted with each batch of filter samples. The recovery of analytes spiked into the LCS will be calculated. Any recovery outside 80-120% indicates a problem with the analytical system and requires investigation to correct the problem before more samples are extracted and analyzed.

12.0 REFERENCES

12.1 Determination of Inorganic Anions by Ion Chromatography, *Test Methods for Evaluating Solid Waste (SW-846)*, U.S. EPA, Third Edition, SW-846 Method 9056, September 1994.

12.2 Quality Assurance Project Plan for the Quality Assurance Laboratory Role in the Particulate Matter ($PM_{2.5}$) Chemical Speciation Project

13.0 APPENDIX A

13.1 Appendix A presents the extraction log form (see Figure 1) which must be completed to document the extraction of all samples prepared for analysis of anions by ion chromatography at NAREL.

Figure 1

Sample Extraction Worksheet Ion Chromatography US EPA/NAREL

Batch number: _____ Date Started: _____ Analyst: _____

Extraction solvent: ☐ Barnstead DI water ☐ Other (describe): _____

Sonication bath: ☐ Yes ☐ No Elapsed sonication time: _____ minutes Max/min bath temperature: _____ / _____ °C

Mechanical shaker: ☐ Yes ☐ No Shaker speed: _____ cycles/min Max/min room temperature: _____ / _____ °C

Sample ID	Sample Description	Solvent added (mL)	Spike added		Comments
			(code)	(mL)	

Spike Solutions

Code	Notebook Number	Code	Notebook Number	Code	Notebook Number
A		B		C	

Reviewed by: _____

14.0 APPENDIX B

14.1 Appendix B presents the injection log forms (see Figure 2 and Figure 3) which must be completed to document the analysis of anions by ion chromatography at NAREL. Instructions for completing these forms follow.

14.2 INJECTION LOG INSTRUCTIONS. Daily information in this injection log is spread over two pages. Thus, each day has an A and a B page. Most of the fields will be self explanatory. Some headings that may not be obvious are as follows.

14.3 Data Archive (Under Miscellaneous Information): *Dionex PeakNet*© software running on a dedicated PC acquires and processes IC data stored on the local hard disk. Use this "Data Archive" field to record the pathway to data files described elsewhere on the logbook page under the "DATA METHOD" and "FILE ID" columns. Please note that most of the file pathway has already been printed, but the pathway needs completion with a manual entry. For example, A052400a would represent a directory for anions (A...) acquired on May 24, 2000, (...052400...) that was the first batch of the day (...a), and the complete pathway to this directory would be C:\PeakNet\data\anions\A052400a.

14.4 Col. Pressure and Det. Baseline (Under Miscellaneous Information): Pressure at the inlet of the guard column and detector baseline should be recorded. These values are conveniently available on the instrument display panel. Most of the other instrument conditions are available in the archived snapshot of the *PeakNet*© method.

14.5 I.S. (Under Standards): The laboratory notebook number of the Internal Standard solution should be recorded along with the volume spiked into each mL of sample. Other standards (up to seven more) should be documented by recording the laboratory notebook number in this region of the logbook page.

14.6 ANALYTICAL METHOD: This is the approved protocol that has been agreed upon by the client. (e.g. PM2.5 SOP, SW-846 9056, EPA 300.0, etc.)

14.7 SOFTWARE METHOD: This is the *Dionex PeakNet*© method used to acquire and process IC data. A snapshot of this method is archived along with the associated data files.

14.8 FILE ID: The data file naming conventions are as follows. A root name such as C052400 is provided to the software to represent Anions (A...) acquired on May 5, 2000 (...052400...) as the first batch of the day (...a). The system will supply the underline character (_), a three-digit counter, and the .DXD file extension as a suffix to the file name root such that the first file for the batch has a file name of A052400_001.DXD.

14.9 DF: This is the Dilution Factor.

14.10 COMMENTS: These are comments that aren't covered by one of the other fields. Anomalies or trends should be noted here.

14.11 G/R: This signifies useable data. An IC sample is either a good injection (G) or needs re-analysis (R).

Figure 2

1A **U.S. EPA/NAREL Anions by IC LOGBOOK**

MISCELLANEOUS INFORMATION

Analyst		**Data Archive**	**C:\PeakNet\data\anions**
Date		**Comments**	
Col. Pressure	**psig**		
Det. Baseline	**µS**		

INJ	PROJECT	NAREL SDG	NAREL SAMPLE ID	CLIENT SAMPLE ID	ANALYTICAL METHOD	MATRIX
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						

Figure 3

U.S. EPA/NAREL Anions by IC LOGBOOK **1B**

STANDARDS

Eluent		**Std C**	
I.S.		**Std D**	
Std A		**Std E**	
Std B		**Std F**	

SOFTWARE METHOD	FILE ID	EXT. DATE	DF	COMMENTS	G/R	INJ
.met	.DXD					1
.met	.DXD					2
.met	.DXD					3
.met	.DXD					4
.met	.DXD					5
.met	.DXD					6
.met	.DXD					7
.met	.DXD					8
.met	.DXD					9
.met	.DXD					10
.met	.DXD					11
.met	.DXD					12
						13
						14
						15

REVIEWED BY _____

15.0 APPENDIX C

15.1 Appendix C presents an example chromatogram of the mid-level calibration standard (see Figure 4). The instrument was equipped with a 100-µL injection loop. The sample contained fluoride (1 ppm), chloride (1 ppm), nitrite (1 ppm), selenite internal standard (10 ppm), nitrate (2 ppm), sulfate (2 ppm), and selenate internal standard (10 ppm). The flow rate of the eluent was 1 mL/minute which produced a column head pressure of 1600 psig. The current for the Self Regenerating Suppressor (SRS) was set at 100 mA, and the detector temperature was set at 35 °C.

Figure 4